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TCL, SQL requirements and QA/QC measures are listed in Section 3.0. All detected herbicides will undergo confirmatory analysis on a chemically dissimilar second column.

## 7.1.6 PCDD/PCDF Analyses

PCDDs and PCDFs will be analyzed by Method 1613 Revision A (1613A) April, 1990 as specified in Table 7-1. A copy of the analytical method appears in Appendix E of this QAPP. This method uses high resolution gas chromatography/high resolution mass spectroscopy (HRGC/HRMS) in the selected ion monitoring (SIM) mode for the detection and quantitation of PCDDs (tetra through octachlorinated homologues) and PCDFs (tetra through octachlorinated homologues) at part-per-trillion (ppt) concentrations for soil/sediment samples and pg/L concentrations for aqueous samples. EPA Method 1613A is considered preferable to the SW-846 draft Method 8290 as 1) SW-846 draft Method 8290 is not promulgated, 2) Method 1613A utilizes additional labeled internal standards for all 2,3,7,8-substituted isomers except the octachlorinated dibenzofuran (OCDF) providing more accurate and reliable results and 3) historic PCDD/PCDF data from past sampling events (e.g., 1989 through 1993) in the Passaic River, Newark Bay, and its tributaries by EPA-Region II, Maxus and others used Method 1613A for PCDD/PCDF analysis (e.g., PCDD/PCDF data from surface sediment samples analyzed by Midwest Research Institute and sediment samples analyzed by Pacific Analytical, Inc. for the NOAA/USFWS/EPA study and supplied by Eric Stern of EPA-Region II). The compounds to be determined by this method are listed in Table 3-6.

## 7.1.7 Total Extractable Petroleum Hydrocarbons (TEPH)

TEPH will be analyzed by the California Leaking Underground Fuel Tank (CA LUFT) method (May, 1988) as specified in Table 7-1 with the following specific requirements. The extraction solvent used will be methylene chloride and soil samples will be extracted

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using a soxhlet extraction while aqueous samples will be extracted either with a separatory funnel or by a continuous liquid-liquid extraction. The analytical results reported will include those hydrocarbons within the  $C_9$  to  $C_{28}$  range. Integration of the chromatographic peaks for these hydrocarbons will include all peak areas above the baseline. A surrogate compound such as pentacosane will be added to each sample. A copy of the CA LUFT method appears in Appendix G of this QAPP.

## 7.1.8 Metals and Cyanide Analyses

Concentrations will be determined using graphite furnace atomic adsorption (GFAA) for arsenic (Method 3050A, Revision 1, July 1992/Method 7060, Revision 0, September 1986), selenium (Method 3050A, Revision 1, July 1992/Method 7740, Revision 0, September 1986), thallium (Method 3050A, Revision 1, July 1992/Method 7841, Revision 0, September 1986), and lead (Method 3050A, Revision 1, July 1992/Method 7421, Revision 0, September 1986); cold vapor atomic absorption (CVAA) for mercury; inductively coupled plasma emission spectroscopy (ICP) (Method 3050A, Revision 1, July 1992/Method 6010A, Revision 1, July 1992) for the remaining metal target analytes; and titrimetric or colorimetric for cyanide (Method 9013, Revision 0, July 1992/Method 9010A, Revision 1, July 1992) as specified in Table 7-1. Copies of the extraction methods and analytical methods for metals and cyanide are included in Appendix H and F respectively, of this QAPP. Post-digestion spikes will be required for all analytes analyzed by GFAA. For the GFAA analyses, the laboratory will follow the procedures summarized in the Furnace Atomic Adsorption Analysis Scheme (Method of Standard Additions "(MSA) Tree") for quantitation as specified in the Contract Laboratory Program Statement of Work for Inorganics Analysis (ILM 02.0). The specific target analytes, SQL requirements, and QA/QC measures are listed in Section 3.0.

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## 7.1.9 Other Analyses

The laboratory analysis method for TOC will be the modified Lloyd Khan Method, July 1988; and the method for TSS will be Method 160.1, 1971. The analysis methods for the radiochemical parameters (<sup>210</sup>Pb, <sup>137</sup>Cs, <sup>7</sup>Be), will be as specified in Table 7-1. Copies of the analytical methods for TOC and TSS appear in Appendix I of this QAPP. Method summaries for the lead-210, the cesium-137, and beryllium-7 analyses appear in Appendixes J and K, respectively. SQL requirements (except for the radiochemical parameters) are listed in Section 3.0.

### 7.2 GEOTECHNICAL TESTS

Geotechnical and physical tests for sediment samples to provide information for the sediment mobility modeling and feasibility study are specified in Table 7-2. Copies of the procedures are included as Appendix L.

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TABLE 7-1
ANALYTICAL PROCEDURES

		Extraction and Analysis Method <sup>1,2</sup>	
Parameter <sup>3</sup>	Technique <sup>4</sup>	Water	Soil/Sediment
Volatile Organics	GC/MS	INC/8260	INC/8260
Semivolatile Organics	GC/MS	3520A/8270A	3550A/8270A
Pesticide/PCBs	GC	3520/8081	3550/8081
Chlorinated Herbicides	GC	INC/8150A	INC/8150A
PCDDs/PCDFs	HRGC/HRMS	INC/1613A	INC/1613A
TEPH	GC	INC/CA LUFT	INC/CA LUFT
Metals Arsenic Lead Selenium Thallium Mercury	ICP GFAA GFAA GFAA CVAA	3010/6010A INC/7060 3020A/7421 INC/7740 3020A/7841 INC/7470	3050A/6010A 3050A/7060 3050A/7421 3050A/7740 3050A/7841 INC/7471
Cyanide	Titration/Colorimetric	INC/9010A <sup>5</sup>	9013/9010A <sup>5</sup>
Total Organic Carbon (TOC)	Carbonaceous Analyzer	INC/9060	INC/Lloyd Kahn <sup>6</sup>
Total Suspended Solids (TSS)	Gravimetric	160.2	N/A
Radiochemical: Lead-210 Cesium-137	Alpha Spectroscopy Gamma-Ray Spectroscopy Gamma-Ray Spectroscopy	N/A N/A	Appendix J Appendix K
Beryllium-7		N/A	Appendix K

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# **TABLE 7-1** (Concluded)

- The 3000 through 9000 methods are from USEPA SW-846 "Test Methods for Evaluating Solid Waste", Third Edition, November 1986 including promulgated Update I and proposed Update II. The method for TSS is from "Methods for Chemical Analysis of Water and Wastes", USEPA, EPA600/4/79/020, revised March 1983. The CA LUFT method is from State of California "Leaking Underground Fuel Tank Field Manual, May 1988. California Department of Health Services. The method for PCDDs/PCDFs is USEPA Method 1613: Tetra through Octa Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision A, April 1990. Copies of the extraction methods, analytical methods, and method summaries are included in Appendixes A through K of this QAPP.
- <sup>2</sup> 'INC' indicates that the sample preparation method is included in the analytical method.
- <sup>3</sup> PCBs = polychlorinated biphenyls; PAHs = polynuclear aromatic hydrocarbons; PCDDs/PCDFs = polychlorinated dibenzo-p-dioxins/polychlorinated dibenzofurans; TEPH = total extractable petroleum hydrocarbons.
- GC/MS = gas chromatography/mass spectroscopy; GC = gas chromatography; HRGC/HRMS = high resolution gas chromatography/high resolution mass spectroscopy; ICP = inductively coupled plasma emission spectroscopy; GFAA = graphite furnace atomic adsorption; CVAA = cold vapor atomic adsorption.
- <sup>5</sup> Method 9012 (automated UV, Colorimetric) shall also be acceptable for cyanide analyses.
- <sup>6</sup> Lloyd Kahn TOC method, as modified by EPA, is included in Appendix I.

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### **TABLE 7-2**

## GEOTECHNICAL AND PHYSICAL TESTS FOR SOIL/SEDIMENT SAMPLES<sup>1</sup>

Procedure	Method Number <sup>2</sup>
Compressibility	ASTM D2435-90
Grain Size Distribution	ASTM D422-63
Dry Density	ASTM D2937-83
Wet Bulk Density	ASTM D2937-83
Moisture Content	ASTM D2216-90
Atterberg Limits	ASTM D4318-84

 $<sup>^{\</sup>rm 1}$  Copies of the ASTM procedures to be used are included in Appendix L of this QAPP.  $^{\rm 2}$  ASTM - American Society for Testing and Materials.

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8.0

### DATA REDUCTION, VALIDATION AND REPORTING

The analytical data generated by the laboratory(ies) will be checked for accuracy, precision, and completeness. The chemical data production and validation process for this project will consist of data generation, reduction, and two levels of review.

### 8.1 ANALYTICAL LABORATORY DATA REVIEW PROCESS

The first level of review, which may contain multiple sublevels, will be conducted by the analytical laboratory Data Reviewer who has the initial responsibility for the correctness and completeness of the data. The laboratory data reviewer will evaluate the quality of the analytical data based on an established set of laboratory guidelines and this QAPP. This person will review the data packages to confirm the following:

- C Sample preparation information is correct and complete.
- C Analysis information is correct and complete.
- C The appropriate SOPs have been followed.
- C Analytical results are correct and complete and all soil/sediment results are reported on a dry weight basis.
- QC samples are within established control limits.
- C Blanks are within appropriate QC limits.

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- C Analytical results for QC sample spikes, sample duplicates, initial and continuous calibration verifications of standards and blanks, standard procedural blanks, laboratory control samples, and ICP interference check samples are correct and complete.
- C Tabulation of reporting limits related to the sample are correct and complete.
- C Special sample preparation and analytical requirements have been met.
- C Documentation is complete (all anomalies in the preparation and analysis have been documented; holding times are documented).

The laboratory will perform the in-house analytical data reduction and QA review under the direction of the laboratory data review supervisor. The laboratory is responsible for assessing data quality and advising the CPM of any data which were rated "preliminary" or "unacceptable," or other notations which would caution the data user of possible unreliability. Data reduction, QA review, and reporting by the laboratory will be conducted as follows:

- Raw data produced by the analyst are processed and reviewed for attainment of QC criteria as outlined in this QAPP and/or established EPA methods for overall reasonableness.
- C The data reviewer will check for entry errors from all manually entered sample data and will check for transfer errors for all data electronically uploaded from the instrument output into the software packages used for calculations and generation of report forms and will decide whether any sample re-analysis is required.

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C The laboratory will review initial and continuing calibration data, and calculation of response factors, surrogate recoveries, MS/MSD recoveries, post-digestion spike

recoveries, internal standard recoveries, LCS recoveries, and sample results.

Upon acceptance of the preliminary reports by the data reviewer, the Laboratory QA Officer or designee will review and approve the data packages prior to the final reports

being generated.

The data reduction and the QC review steps will be documented, signed, and dated by the analyst.

The laboratory will prepare and retain full analytical and QC documentation. The laboratory will report the data as a group of 20 environmental samples or fewer, along with QC supporting data. These groupings of samples (Sample Delivery Groups) will be assigned by the field sample collection and processing team. For each analysis type, other than the PCDD/PCDF analyses, the laboratory will, at a minimum, provide the hard copy information listed below in each analytical data package submitted using CLP-equivalent forms. These forms shall contain all of the information contained on the CLP forms that is pertinent to the analytical method requirements.

- Chain-of-Custody, cover sheet listing the samples included in the report and narrative comments describing problems encountered in analysis and identification of analyses not meeting QC criteria, including holding times
- C Analytical results for QC sample spikes, sample duplicates, initial calibration and continuing calibration verifications of standards and blanks, standard procedural blanks, and laboratory control samples

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- C Tabulated results of compounds identified and quantified, dilution factors, and the reporting limits for all analytes; with analytes detected below the SQL reported as non-detects at the SQL.
- Summary reports for initial and continuing calibrations listing relative response factors (RRFs) and percent relative standard deviations (%RSDs) for organics and percent recoveries and true values for inorganics, MS/MSD percent recoveries or MS/duplicate for inorganics, spike amounts, and RPDs, surrogate spike percent recoveries and spike amounts (if applicable), laboratory blank results, and a method blank summary listing method blanks and associated samples
- Raw data system printouts (or legible photocopies) and chromatograms (identifying sample identification, date of reported analysis, parameters analyzed), for samples, initial calibration, calibration verifications, method blanks, any reported sample dilutions, sample duplicates, spikes, and control samples; sample spiking levels; preparation/extraction logs and run logs

For organic analyses, the data packages will include MS/MSD, surrogate spike recoveries (if applicable), chromatograms, GC/MS spectra (if appropriate to the method), and computer printouts for reported analyses and associated QC data.

For the PCDD/PCDF analyses, the data packages will include, at a minimum, the following:

- a. PCDD/PCDF Analysis Data Sheet (Form 1a)
- b. PCDD/PCDF Confirmation Analysis Data Sheet (Form 1b) if applicable
- c. Cleanup Standard Recoveries (Form 2)
- d. Initial Calibration Relative Responses (Form 3a)